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METHODS FOR IDENTIFICATION OF APPLICATION CLASSES OF DYES ON TEXTILE MATERIALS

PART II WOOL, SILK AND OTHER PROTEIN FIBRES

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
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PART II WOOL, SILK AND OTHER PROTEIN FIBRES

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METHODS FOR IDENTIFICATION OF APPLICATION CLASSES OF DYES ON TEXTILE MATERIALS

PART II WOOL, SILK AND OTHER PROTEIN FIBRES

0. FOREWORD

0.1 This Indian standard (Part II) was adopted by the Indian Standards Institution on 30 December 1968, after the draft finalized by the Dyestuffs Sectional Committee had been approved by the Textile Division Council.

0.2 The methods of identification of dyes on wool, silk and other protein fibres prescribed in this standard, are simple and are meant for rapid identification of application classes of dyes. Successful identification of application classes of dyes depends on experience and familiarity of the dyestuffs. The knowledge of a fibre eliminates dyes which are not applicable to the particular fibre and the identification is made somewhat easier.

0.3 The methods prescribed here do not involve the use of microscope, however, its use is not precluded if desired. In general, the identification of application classes of dyes is not dependent on any single test and final confirmation of the identity of an unknown class of dye should preferably be made by comparison with an authentic sample.

0.4 Considerable assistance has been derived from the following:

ELLIS CLAYTON. Identification of dyes on textile fibres. 1963. Ed 2. The Society of Dyers and Colourists, Bradford, UK.

KOCH (P). Microscopic and chemical testing of textiles. 1963. Chapman and Hall Ltd, London.

Identification of dyestuffs on textiles. Textile Industries Journal 122, 1; 1958; 101-104.

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1. SCOPE

1.1 This standard (Part II) prescribes methods for identification of application classes of dyestuff on wool, silk and other protein fibres.

1.1.1 The methods are applicable to types of dyes normally used for dyeing and printing wool, silk and other protein fibres.

2. PREPARATION OF TEST SPECIMEN

2.1 If the sample under test is fibre or yarn, take a tuft of fibre or yarn of about 3 cm in length.

2.2 If the sample under test is fabric, take a 3 × 3 cm test piece.

NOTE 1 — In case of multi-coloured woven fabric, the different colours on yarn should be identified separately.

NOTE 2 — In case of dyed and printed fabrics, the specimens should be taken from different coloured portions of the sample.

2.3 To remove finishing materials, if any, present in textiles, the sample should be treated twice with 1 percent hydrochloric acid at boil for 5 minutes and washed.

3. REAGENTS

3.0 **Quality of Reagents** — Unless specified otherwise, pure chemicals shall be employed in tests and distilled water (*see* IS: 1070-1960*) shall be used where the use of water as reagent is intended.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the test results.

3.1 **Acetic Acid** — (a) 5 percent (*w/v*); and (b) glacial.

3.2 **Ammonium Hydroxide Solution** — prepared by diluting 1 ml of concentrated ammonia (sp gr 0·90) to 100 ml with water.

3.3 **Sodium Chloride** — solid.

3.4 **Sulphuric Acid** — 10 percent (*w/v*).

3.5 **Sodium Carbonate** — (a) 5 percent solution (*w/v*), and (b) solid.

3.6 **Sodium Nitrate** — solid.

3.7 **Sodium Hydroxide Solution** — 1, 5, 10 and 30 percent solutions.

3.8 **Sodium Hydrosulphite** — (a) 10 percent (*w/v*) freshly prepared, and (b) solid.

3.9 **Sodium Bisulphite** — 30 percent (*w/v*).

3.10 **Rectified Spirit** — *See* IS: 323-1959†.

3.11 **Hydrogen Peroxide** — 3 percent (*w/v*) (10 volumes).

*Specification for water, distilled quality (*revised*).
†Specification for rectified spirit (*revised*).

3.12 Hydrochloric Acid — (a) prepared by diluting 15 ml of concentrated hydrochloric acid to 100 ml, and (b) concentrated (sp gr 1·16).

3.13 Ethylenediamine — sp gr 0·97, b.p. 117°C.

3.14 Ether

3.15 Bleached Cotton

3.16 Scoured Wool (Undyed)

3.17 Dimethyl Formamide — (a) 50 percent (*w/v*), and (b) concentrated (b.p. 152° to 154°C).

3.18 Tannin Reagent — prepared by dissolving 10 g of tannic acid and 10 g of anhydrous sodium acetate in 200 ml of water.

3.19 EDTA — Glycerine Solution — 4 percent (*w/v*) solution of disodium salt of ethylenediamine tetra acetic acid in glycerine.

3.20 Paraffin Wax

3.21 Blank Vat Solution — prepared by dissolving 15 g of sodium hydroxide and 20 g of sodium hydrosulphite in sufficient water and made to 1 litre.

4. PROCEDURE

4.1 For identification of dyes on fibres, follow the procedure given in Appendix A.

NOTE — While identifying the dyes used for dyeing pale shades, it is advisable to use large specimen and large quantities of reagents and to concentrate the extract before making the test.

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APPENDIX A

(*Clause* 4.1)

TESTS FOR IDENTIFICATION OF APPLICATION CLASSES OF DYES ON WOOL, SILK AND OTHER PROTEIN FIBRES

The test specimen is successively treated with (a) 50 percent dimethylformamide, (b) concentrated dimethylformamide, and (c) a mixture of glacial acetic acid and rectified spirit (1 : 1, *v/v*) at boil for 3 to 4 minutes with intermediate washing with water and squeezing.

If dyes of group I are absent, treat the test specimen with 0·1 ml of glacial acetic acid and warm. Add 5 ml of water.

If dyes of groups I and II are absent, take a fresh test specimen and add to it 5 to 10 ml of 1 percent ammonium hydroxide solution. If extract is coloured, remove the stripped specimen and divide the extract into two portions. (a) Add to the first portion 30 mg of sodium chloride and 10 to 30 mg each of bleached cotton and secured wool, and boil. (b) Neutralize the other portion by adding 10 percent sulphuric acid and few drops in excess. Add bleached cotton and secured wool and boil.

If dyes of groups I, II and III are absent, take a test specimen (about 5 g) and ash it completely in a porcelain crucible. Add 200 mg of flux (equal parts of sodium carbonate and sodium nitrate) and fuse.

If dyes of groups I to IV are absent, take a test specimen (about 200 to 300 mg) and add 2·5 ml of 10 percent sodium hydroxide and boil until it is dissolved. Add 25 to 30 mg of sodium hydrosulphite, 20 to 50 mg of sodium chloride and 10 to 15 mg of bleached cotton. Keep near boil for 2 minutes and cool. Take out the cotton and place it on filter paper for 1 to 2 minutes and oxidize with sodium nitrate and acetic acid solution.

GROUP I	GROUP II	GROUP III	GROUP IV		GROUP V	
No stripping or partial stripping of dye REACTIVE DYES	The acetic acid extract is distinctly coloured BASIC DYES	Cotton is dyed in first portion DIRECT DYES	Wool is dyed in second portion ACID DYES (*If Direct and Basic Dyes Absent*)	Test for the Presence of Metal (Cr or Co present) *Metal complex or Mordant dyes (chrome)*	Cotton is dyed. In case of very pale dyeings it may be necessary to re-dye the cotton in 2 to 4 successive solutions stripped from the original dyeing VAT DYES	Cotton is undyed and colour is destroyed AZOIC DYES
				Strip the dye in hot 1 percent ammonium hydroxide. After cooling acidify the solution with hydrochloric acid. Shake up the extract with ether		
				Ether is coloured *Metal Complex Dyes*	Ether is not coloured *Mordant Dyes (Chrome)*	

ADDITIONAL TESTS

1) *Basic Dyes*

i) Take a test specimen. Add to it 1 ml of glacial acetic acid and warm. Add 5 ml of water. To the extract add tannin reagent. A coloured precipitate is obtained.

ii) Take a test specimen, boil it with rectified spirit; a coloured extract is obtained.

iii) Take a test specimen, boil it in 2 ml glacial acetic acid. Add 30 percent sodium hydroxide till the solution is alkaline. A change in colour or complete decolourization is obtained. Acidify the solution with 5 percent acetic acid. The original colour is restored.

2) *Direct Dyes*

Take a test specimen and boil it with 5 percent sodium carbonate solution for half a minute in presence of few pieces of bleached cotton. The cotton is stained. The stained cotton is practically unaffected on treatment with 1 percent ammonium hydroxide solution at boil.

NOTE 1 — In case of silk dyeings 5 to 10 percent of sodium hydroxide should be used instead of sodium carbonate solution.

NOTE 2 — In the above test, certain dyes which are closely allied to substative azo dyes in chemical structure, stain the cotton to a comparatively slight extent. Identification of such dyeings may be achieved by boiling the specimen in 1 percent sodium hydroxide solution when dull yellowish colour is obtained. This yellowish colour may also be obtained on boiling the dyeings with 30 percent sodium bisulphite solution.

3) *Acid, Metal Complex and Mordant Dyes (Chrome)*

i) Take a test specimen. Boil it with dimethylformamide. Bleeding indicates *acid dyes*, slight bleeding indicates *metal complex dyes*. No bleeding indicates *mordant dyes (chrome)*.

ii) Heat a test specimen in solution of EDTA in glycerine. At 140°C: No change — *Acid and mordant dyes*. Rapid change (in 1 to 2 minutes) — 1 : 1 *metal complex dyes* (acid dyeing). Slow change (in 20 minutes) — 1 : 2 *metal complex dyes* (neutral dyeing). At 160°C: No change — Acid Dyes.

iii) Take a test specimen. Boil it with dilute hydrochloric acid [*see* **3.12**(a)]. Take out the specimen and wash and treat it with 10 percent sodium hydrosulphite solution. The colour is destroyed.

iv) Most of the after chrome dyes are not stripped. This fact should be taken as a clue for mordant dyes (chrome).

4) *Vat and Azoic Dyes*

i) Warm some paraffin wax in a white porcelain crucible until faint vapours appear. Take a test specimen and hold it in molten wax for about a minute. Remove the specimen. After cooling any staining of paraffin wax is readily seen against white background of the porcelain.

ii) Take a test specimen and treat it with a blank vat solution at 60°C in a test-tube. Oxidize the specimen with hydrogen peroxide (3 percent, *w/v*).

a) Change in colour and original colour restored on oxidation — *vat dyes*.

b) Test solution becomes yellow. Colour of the pattern usually remains unchanged. If the colour is changed original colour not restored on oxidation — *azoic dyes*.

iii) Take a test specimen. Warm it with ethylenediamine. Add aqueous solution of sodium hydrosulphite [*see* **3.8**(a)] to ethylenediamine extract. The coloured extract is decolourized readily and permanently.

NOTE — Most azoic dyeings on wool and silk yield slimy residues of the same intense colours as the original dyes, on boiling dyeings in 5 and 10 percent sodium hydroxide solution respectively (distinction from mordant dyes). Most yellow dyeings and prints change to orange or red colours.